63-3-4

AZUSA PLANT

STRUCTURAL MATERIALS DIVISION

INVESTIGATION OF STRESS-CORROSION CRACKING OF HIGH-STRENGTH ALLOYS

A Report To

FRANKFORD ARSENAL

Contract DA-04-495-ORD-3069

Report No. L0414-01-22 / May 1963 / Copy No. 12

AEROJET
GENERAL TIRE
GENERAL

This is the twenty-second in a series of informal monthly progress reports submitted in partial fulfillment of Contract DA-04-495-ORD-3069. It constitutes the sixth monthly progress report for the one-year continuation of the original two-year program.

This report covers the period 1 February through 28 February 1963. It was written by R. B. Setterlund who was supervised by A. Rubin.

AEROJET-GENERAL CORPORATION

P. L. Jordan

P. L. Jordan, Head
Metallics and Refractories Dept.
Structural Materials Division

NOTE: The information contained herein is regarded as preliminary and subject to further checking, verification, and analysis.

I. OBJECTIVES

The objectives of this program are outlined below:

A. Investigation of the stress-corrosion cracking characteristics of at least three new high-strength alloys of interest for rocket motor case applications. These alloys are 6Al-4V titanium, 18%-nickel maraging steel, and 20%-nickel maraging steel, in addition to limited testing of vacuum-melted 9Ni-4Co steel.

B. Study of the environmental parameters that could affect the rate and extent of stress-corrosion cracking.

C. Determination of the effect of material parameters (composition, strength level, welding, and microstructure) on stress-corrosion susceptibility.

D. Continuation of the evaluation of protective coatings and other techniques for preventing stress-corrosion cracking.

II. SUMMARY

Results obtained to date indicate that the 6Al-4V titanium alloy is immune to stress-corrosion cracking under the test conditions of this program in both the annealed and the quenched-and-aged conditions. Preliminary test results indicate that welded 6Al-4V is also immune to stress-corrosion cracking.

The 20%-nickel maraging steel was found to be highly susceptible to stress-corrosion cracking in the annealed-and-aged condition. When the same material was cold-worked before aging, however, the resistance to stress-corrosion cracking was greatly increased.

The 18%-nickel maraging steel was also found to be susceptible to stress-corrosion cracking. While this material was found to have a longer time-to-failure than the 20%-nickel steel in distilled water, salt water, and 140°F moisture-saturated

air, it showed some failures in tap water, chromate solution, and soluble oil solutions where the 20%-nickel steel was not affected. As with the 20%-nickel steel it was found that prior cold working decreased the stress-corrosion cracking susceptibility.

Fifteen different coating systems designed to prevent stress-corrosion cracking are under evaluation using H-11 steel as the base material, heat treated to give it a high failure susceptibility. Two chromate-bearing epoxy systems show a promising capability for preventing stress-corrosion cracking.

III. WORK PROGRESS

A. INTRODUCTION

Since the initiation of the original test program, two years ago, to investigate the stress-corrosion cracking characteristics of high-strength alloys, a number of new high-strength steels have been receiving increased attention for use in constructing rocket motor cases. The third-year test program is directed to the study of three of these new alloys, as well as of one titanium alloy presently being used for the same application.

The test environments, substantially the same as those evaluated in the original two-year investigation, are as follows: (1) distilled water; (2) tap water; (3) salt water; (4) sodium dichromate-inhibited water; (5) soluble oil-inhibited water; (6) air; (7) high humidity atmosphere; (8) trichloroethylene; (9) cosmoline; and (10) solid propellant. These are considered to be environments representative of those to which rocket motors cases would normally be exposed during fabrication, processing, and storage. One additional environment is included in the new program, that of sea-coast atmospheric exposure.

The test methods being used in this investigation employ bent-beam U-bend, and center-notched specimens. Evaluation of results includes microstructural studies, using both standard metallographic and electron microscopic techniques, to attempt to associate the failure mechanism with specific microstructural characteristics of the materials.

An evaluation of protective coatings and surface treatments to prevent stress-corrosion cracking is also being conducted.

B. PROGRAM STATUS

Table 4 lists the test results obtained with the 6Al-4V titanium alloy. In addition to the tests shown, the alloy is also being tested in the annealed-and-welded condition (Test 6-W, Table 1). Results to date have shown this alloy to be completely immune to stress-corrosion cracking under all the conditions of this program. This immunity was indicated in the bent-beam tests, as well as in the more sensitive center-notched, constant-load tensile tests which were performed.

Test results, to date, with the maraging steels, involving bent-beam and center-notched specimens are shown in Table 5. Since the compiling of these data, three more alloy conditions, H-2, I-2, and I-4 (Table 1) have been started. However, since the exposure times are still short, these data will be shown in the quarterly report. As indicated in Table 5, both the 20%- and 18%-nickel maraging steels showed some failures when tested in both the annealed-and-aged and cold-worked-and-aged conditions. There was, however, a wide variation in susceptibility to cracking in different environments. For example, the annealed-and-aged 20%-nickel steel failed more rapidly in distilled water and salt water than the annealed-and-aged 18%-nickel steel; yet the latter material failed in tap water, chromate solution, and soluble oil solution where the 20%-nickel steel was immune to cracking. When either the 18%- or 20%-nickel steels were cold-worked before aging, it was found that susceptibility to stress-corrosion cracking was reduced and, at the same time, the mode of cracking changed from intergranular failures to possible cracking along slip planes. This effect was most clearly shown with the 20%-nickel steel. Photomicrographs of both types of failures were included in the previous quarterly report, and a more complete analysis will be made in the next quarterly report.

Both the 18%- and 20%-nickel maraging steels have been successfully welded using an automatic TIG process without filler metal. We expect to have the welded 20%-nickel alloy in test the last week of April with the 18%-nickel steel to follow in early May.

The results of the coating evaluation program are shown in Table 6. Fifteen different coatings are under test in both an aerated salt-water environment and a high-humidity atmosphere. Testing has also started on an outdoor seacoast test rack. Data from these tests will be included in the quarterly report. Based on tests to date, two inhibited-epoxy coatings, 454-1-1 and 463-1-5, show the most promise for protecting a susceptible base metal from stress-corrosion cracking.

IV. FUTURE WORK

Work will be continued along the guidelines of the master plan shown in Table 1. Both bent-beam and center-notched specimens will be immersed as required to fulfill this schedule.

It is expected that all the remaining maraging steel samples, including welded specimens, will be in test by 1 May. The 0.25 to 0.30% carbon grade of the 9Ni-4Co alloy has been received, and some limited testing of this alloy will soon be started.

Outdoor seacoast exposure tests have been started. Results from these tests will be included in the next quarterly report.

Metallographic sections of selected cracked samples have been prepared and photographed. In addition, the cracking process is being studied by means of the electron microscope, utilizing fracture replicas. The intention is to attempt to define the mode of failure and, if possible, associate the failure process with microstructural characteristics of the materials. Both photomicrographs and electron microscope fractographs will be presented in the next quarterly report.

V. BUDGET

The expenditure rate for the month of February was 240 hours leaving a total of 960 hours to be expended on the remainder of the program.

TABLE 1

MASTER PLAN - BENT BEAM STRESS-CORROSION TESTS

Alloy	Processing Condition (Titanium Content of Maraging Steel Shown)	Strength Level, 0.2% Offset Yield (psi)	Specimen Code	Test Environment: Distilled Water	Tap Water	3% NaCl Solution	0.25% Sodium Dichromate Solution	4% Soluble Oil Solution	High Humidity	Trichloro-ethylene	Cosmoline	Solid Propellant	Ambient Air	Sea Coast Atmosphere	Total
6Al 4V titanium	Annealed	138,000	G-1	3*	3	3	3	3	3	3	3	3	-	3	30
	Quenched and Aged	163,000	G-2	3	3	3	3	3	3	3	3	3	-	3	30
	Welded	135,000	G-W	2	2	2	2	2	2	2	2	-	2	2	20
	Total			8	8	8	8	8	8	8	8	6	2	8	80
20%-Nickel Maraging Steel	Annealed and Aged	291,000	H-1	3	3	3	3	3	3	3	3	3	3	3	33
	50% CW and Aged	321,000	H-2	3	3	3	3	3	3	3	3	3	3	3	33
	75% CW and Aged	298,300	H-3	3	3	3	3	3	3	3	3	3	3	3	33
	Welded and Aged	To be tested	H-W	3	3	3	3	3	3	3	3	3	3	3	33
	Total			12	12	12	12	12	12	12	12	12	12	12	132
18%-Nickel Maraging Steel	Annealed & Aged (0.62% Ti)	283,000	I-1	3	3	3	3	3	3	3	3	3	3	3	33
	50% CW & Aged (0.50% Ti)	302,400	I-2	3	3	3	3	3	3	3	3	3	3	3	33
	50% CW & Aged (0.62% Ti)	323,000	I-3	3	3	3	3	3	3	3	3	3	3	3	33
	Annealed & Aged (0.50% Ti)	249,900	I-4	3	3	3	3	3	3	3	3	3	3	3	33
	50% CW & Aged (0.40% Ti)	278,000	I-5	3	-	2	-	-	3	-	-	-	1	2	11
	Annealed & Aged (0.52% Ti)	255,400	I-6	3	-	2	-	-	3	-	-	-	2	3	13
	50% CW & Aged (0.52% Ti)	331,000	I-7	3	-	2	-	-	3	-	-	-	2	3	13
	Annealed & Aged (1.00% Ti)	323,200	I-8	3	-	2	-	-	3	-	-	-	1	2	11
	50% CW & Aged (1.00% Ti)	354,400	I-9	3	-	2	-	-	3	-	-	-	2	2	12
	Welded & Aged (0.50% Ti)	To be tested	I-W	3	3	3	3	3	3	3	3	3	3	3	33
	Total			30	15	23	15	15	30	15	15	15	23	27	225
9 Ni-4 Co Vacuum-Cast Alloy	Aged (0.25-0.30% C)	To be tested	J-1	3	3	3	3	3	3	3	3	3	3	3	33
	Aged (0.40-0.45% C)	To be tested	J-2	3	3	3	3	3	3	3	3	3	3	3	33
	Total			6	6	6	6	6	6	6	6	6	6	6	66
H-11 Steel (Coating Tests)	Application of Various Protective Coatings	-	-	-	-	37	-	-	29	-	-	-	-	26	92
	Total			56	41	86	41	41	85	41	41	39	43	79	595

*Number of replicate tests conducted.

TABLE 2

CHEMICAL ANALYSIS AND MECHANICAL PROPERTIES OF MARAGING STEELS

Supplier	Heat Numbers	C	Mn	P	S	Si	Ni	Co	Mo	Al	Cb	Zr	Ti	Ca	B
		Mill-Certified Analysis (Percent Composition)													
Allegheny-Ludlum	W-24254	0.009	0.09	0.002	0.005	0.06	20.41	-	-	0.29	0.39	0.002	1.40	0.004	0.003
Allegheny-Ludlum	W-24178	0.012	0.01	0.003	0.005	0.01	18.69	8.90	4.92	0.029	-	0.003	0.62	0.006	0.002
Allegheny-Ludlum	477	0.018	0.002	0.006	0.004	0.024	18.29	9.10	4.95	0.089	-	<0.004	0.40	<0.0006	0.003
Allegheny-Ludlum	448	0.029	0.002	0.004	0.008	0.009	18.51	8.46	4.92	0.089	-	<0.004	0.52	<0.0006	0.003
Allegheny-Ludlum	476	0.020	0.002	0.006	0.005	0.014	18.60	9.05	4.90	0.078	-	<0.004	1.00	<0.0006	0.003
Republic	3960502	0.020	0.08	0.007	0.006	0.15	18.48	7.00	4.84	0.21	-	0.035	0.50	Added	.0036

Allegheny-Ludlum Heat Numbers	Percent Cold Reduction	Aging Treatment	Table 1 Code No.	0.2% Offset Y.S. (psi) (Transverse)	U.T.S. (psi) (Transverse)	Percent Elongation (in 2 in.)	Percent Reduction In Area	Notched Tensile Strength (psi)*	Hardness (Rockwell C)
				Mechanical Properties (Aerojet Tests)					
W-24254	0	None		128,500	170,700	7.5	53	-	34
	0	-100°F + 850°F 4 hr	H-1	291,300	302,200	3	17	58,200	54
	50	None		184,000	201,600	5	50	-	39
	50	850°F 4 hr	H-2	321,000	327,100	3	25	-	55.5
	75	None		205,700	220,900	6	46	-	44
	75	850°F 4 hr	H-3	298,300	308,800	2.5	13	31,500	55
W-24178	0**	None		102,000	153,300	14	62	-	30.5
	0**	900°F 3 hr	I-1	283,000	294,000	8	38	179,100	53.5
	50	None		167,700	189,000	3.5	51	-	36.5
	50	900°F 3 hr	I-3	323,800	328,400	1.5	28	113,500	55
477	50	None		169,300	196,900	6.5	40	-	38.5
	50	900°F 3 hr	I-5	278,000	280,700	2	8	158,800	55
448	0	None		105,300	150,300	10	45	-	30.5
	0	900°F 3 hr	I-6	255,400	265,900	5	9	191,600	52
	50	None		175,500	199,800	4.5	47.5	-	38
	50	900°F 3 hr	I-7	331,000	332,500	1.5	7	175,000	55
476	0	None		128,300	174,700	5.5	48	-	36
	0	900°F 3 hr	I-8	323,300	330,000	2.5	27	162,000	56
	50	None		192,200	217,000	2.5	40	-	41
	50	900°F 3 hr	I-9	354,400	354,900	1	1.5	95,400	58

*Tensile tests of fatigue-cracked specimens shown in Figure 3.

**Original cold-worked material which was re-annealed.

Table 2

TABLE 3

CHEMICAL ANALYSIS AND MECHANICAL PROPERTIES OF 6Al-4V TITANIUM

	Chemical Analysis (% Composition)*								
	C	Al	V	O_2	N_2	H_2	Ti	Fe	Other
Aerojet Analysis	0.3	6.1	4.1	0.083	0.014	80 ppm	Bal	0.16	0.18

	Mechanical Properties (Transverse)			
	Yield Strength (0.2% Offset) (psi)	Ultimate Strength (psi)	Elongation (%)	Hardness (R_c)
Annealed				
Mill report	131,900	141,400	12	33.5
Aerojet test	138,000	143,800	14	34
Notched tensile strength**	--	128,500	-	-
1675°F 1 hr, W.Q. Aged 900°F 8 hr				
Aerojet test	162,700	176,800	7	38.5
Notched tensile strength	--	132,000	-	-
Welded				
Aerojet test	131,500***	135,200	9.5	33.0

*Titanium Metals Corporation HT 4141.

**Using as-fatigue-cracked sample of Figure 3.

***Tensile failures in parent metal.

TABLE 4

STRESS CORROSION OF 6Al-4V TITANIUM
IN VARIOUS ENVIRONMENTS

	Condition G-1* Annealed (as received)			Condition G-2 1675°F 1 hr, W.Q., 900°F 8 hr		
		Failure Times			Failure Times	
Environment	Failed/Tested	Mean (hr)	Range (hr)	Failed/Tested	Mean (hr)	Range (hr)
Bent Beam Tests						
Distilled water	0/3**	-	NF1700***	0/3	-	NF1700
Tap water	0/3	-	↓	0/3	-	↓
3% NaCl sol.	0/3	-	↓	0/3	-	↓
0.25% Sodium dichromate	0/3	-	↓	0/3	-	↓
Soluble oil sol.	0/2	-	↓	0/3	-	↓
Cosmoline	0/3	-	↓	0/3	-	↓
High-humidity atmosphere	0/3****	-	↓	0/3	-	↓
Air	0/3	-	NF1700	0/3	-	NF1700
Solid propellant	0/0	-	-	0/0	-	-
Sea-coast exposure	0/0	-	-	0/0	-	-
U-Bend Tests						
High-humidity atmosphere	0/3	-	NF600	0/3	-	NF600
Trichloroethylene	0/0	-	-	0/0	-	-
Sea-coast exposure	0/0	-	-	0/0	-	-
Center-Notch Tests						
Distilled Water	0/2	-	NF100	0/2	-	NF100
3% NaCl sol.	0/2	-	↓	0/2	-	↓
0.25% Sodium dichromate	0/2	-	↓	0/2	-	↓
Soluble oil sol. (4%)	0/1	-	NF100	0/1	-	NF100

*Refers to code letter in Master Schedule, Table 1.

**Indicates no failures of three samples exposed.

***Indicates no failures in 1700 hours exposure.

****Indicates testing not started.

TABLE 5

STRESS-CORROSION CRACKING OF MARAGING STEELS IN VARIOUS ENVIRONMENTS

	20% - Nickel Maraging Steel						18% - Nickel Maraging Steel					
	Material	Condition H-1*		Material	Condition H-3		Material	Condition I-1		Material	Condition I-3	
Test Environments	Failed/ Tested	Failure Time, hours		Failed/ Tested	Failure Time, hours		Failed/ Tested	Failure Time, hours		Failed/ Tested	Failure Time, hours	
Bent Beam Tests		mean	range		mean	range		mean	range		mean	range
Aerated Distilled Water	3/3**	11	10.2 - 18	1/3	1284	1284 - NF2050	3/3	34.5	20.5 - 46.5	4/4	625	440 - 988
Aerated Tap Water	0/3	-	NF2050***	1/3	1510	1510 - NF2050	2/3	350	325 - NF1950	0/3	-	NF1950
Aerated 3% NaCl Solution	3/3	7.3	6 - 8.5	0/3	-	NF2050	3/3	51.5	19 - 100	2/3	1290	1000 - NF1550
Aerated 0.25% Sodium Dichromate	1/3	1	1 - NF2050	0/3	-	NF2050	3/3	117	100 - 150	0/3	-	NF1950
4% Soluble Oil Solution	0/3	-	NF2000	0/3	-	NF2000	3/3	417	400 - 450	0/3	-	NF1950
Cosmoline Immersion	0/3	-	NF2000	0/3	-	NF2000	0/3	-	NF1950	0/3	-	NF1950
140°F Moisture-Saturated Air	3/3	100	22 - 174	3/3	1200	1080 - 1860	3/3	21	20.5 - 21.5	3/3	260	245 - 290
75°F Air, 40% Humidity	0/3	-	NF2000	0/3	-	NF2000	0/3	-	NF1950	0/3	-	NF1950
Solid Propellant	0/0****	-	-	0/0	-	-	0/0	-	-	0/0	-	-
Seacoast Exposure	0/0	-	-	0/0	-	-	0/0	-	-	0/0	-	-
Center-Notch Tests												
Distilled Water	3/3	5.1	4.6 - 6.6	1/3	120.9	120.9 - NF300	3/3	85.3	83.1 - 87.0	2/2	13.2	12.6 - 13.8
3% NaCl Solution	2/2	7.2	6.6 - 7.8	2/2	40.2	34.4 - 46	2/2	20.6	18.0 - 23.2	2/2	5.9	5.0 - 6.9
0.25% Sodium Dichromate	0/2	-	NF200	0/2	-	NF100	1/2	67.9	67.9 - NF200	1/1	33.2	-
4% Soluble Oil Solution	0/1	-	NF200	0/1	-	NF100	0/1	-	NF150	0/0	-	-
Air	0/0	-	-	0/0	-	-	0/0	-	-	0/0	-	-

*Refers to material code letter of Table 1, Master Schedule.

**Indicates three failures out of three samples exposed.

***Indicates no failures in 1270 hours of testing.

****Indicates testing not started.

TABLE 6

EVALUATION OF PROTECTIVE COATINGS ON H-11 STEEL (FOR PREVENTING STRESS-CORROSION CRACKING)

Surface Condition	Coating	140°F Moisture-Saturated Air Failed/Tested	Failure Times, hours mean	Failure Times, hours range	Aerated 3% NaCl Solution Failed/Tested	Failure Times, hours mean	Failure Times, hours range
Surface Ground or Sanded	None (control)	2/2*	64	48 - 70	4/4	1.6	0.8 - 2.5
	Polyurethane	6/6	3500	2830 - 5500	3/3	149	144 - 168
	Inhibited Epoxy 454-1-1	3/3	2720	2590 - 2850	0/0	-	-
	Inhibited Epoxy 463-1-5	3/3	656	400 - 976	0/3	-	NF2000**
	Inhibited Epoxy 463-4-8	3/3	845	289 - 1512	3/3	550	525 - 578
	Epoxy 463-1-5 over 454-1-1	4/4	4000	2590 - 4950	0/4	-	NF5860
	Zinc Silicate, Type 4	2/2	422	147 - 696	2/2	1.2	0.8 - 1.6
	80% Aluminum Epoxy	2/2	30	16 - 45	2/2	100	100 - 100
	70% Titanium Epoxy	2/2	196	136 - 256	2/2	150	140 - 160
Sand Blasted	None	1/1	26.5	26.5	2/2	18.5	14 - 23
	Pure Vinyl	0/2	-	NF600	0/2	-	NF600
	Zinc Silicate Type 4	0/2	-	NF600	2/2	14	10 - 18
	Zinc Silicate Type 4D with Cover Coat	2/2	513	NF422-504	2/2	76.7	1.5 - 152.5
	Inorganic Zinc Type 11	2/2	821	723 - 819	2/2	687	672 - 702
	Epoxy 188 over Inorganic Zinc Type 11	0/2	-	NF1050	2/2	54	42 - 56
	Organic Zinc XL-4-245	2/2	766	742 - 790	2/2	214	27 - 400
	Modified Vinyl System	0/2	-	NF200	0/2	-	NF200

*Indicates two failures out of two samples exposed.

**Indicates no failures in 2000 hours exposure.